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SOLDERABILITY OF TERMINALS & AGING

REPORT NO. 2

CONTRACT NO. DA 36-039-AMC-00008(E)

TASK 702

SECOND QUARTERLY PROGRESS REPORT

1 JANUARY 1963 TO 31 MARCH 1963

U.S. ARMY ELECTRONICS MATERIEL SUPPORT AGENCY

FORT MONMOUTH, N.J.

REPORT PREPARED BY:

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GENERAL ELECTRIC COMPANY

SYRACUSE, N.Y.

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PURPOSE

It is the purpose of this task to further investigate the area of solderability and the aging of finishes to be soldered. The specific areas under this investigation are the solderability of lugs, tabs, stranded wire, etc., and the development of a short time test which will simulate the aging of a lead for six or twelve months. The results of this investigation will be a proposed Military Standard.

ABSTRACT

The objective of this project is to develop proposed test methods for inclusion in Military Standards. These test methods are (1) a simulated short time aging method to duplicate six or twelve months aging of terminations in the field and (2) a solderability test for lugs, tabs, stranded wire, etc.

This report discusses the work done during the second quarter and covers the second phase of each part of this task.

Part I, Phase II is concerned with collecting data on the effects of solderability by exposure to heat, humidity and boiling distilled water; the latter being a re-evaluation of work previously done by others.

Part II, Phase II is concerned with collecting data on new solderability test methods. These all involve flowing a preform but use different heat sources. These include heated flux bath, resistance and induction.

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3. Hoare, W. E. and Britton, S. C. Tinplate Testing, Tin Research Institute, 1960.

4. Symposium on Solder, ASTM Special Technical Publication No. 189, American Society for Testing Materials, 1956.

EXPERIMENTAL WORK

Part I - Aging of Terminals

A. General

The work this quarter has been concerned with short time aging methods. Three of these methods were investigated; boiling distilled water, heat and steam. Of these methods only the steam appears to hold promise of being an acceptable method.

B. Boiling Distilled Water Aging

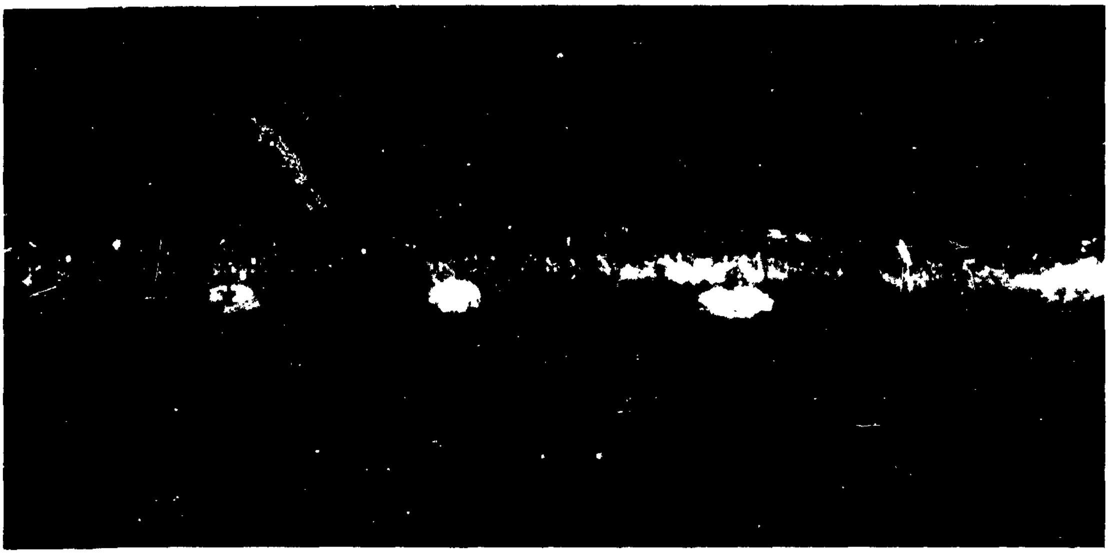
This particular aging method was suggested by Battelle Memorial Institute[1] in 1958 as the one which most nearly simulated natural aging. It is quite simple in that it involves only an immersion of the parts to be tested for one hour in boiling distilled water. However, due to various reasons, it was never adopted for use in a military standard.

In two sets of experiments 190 samples (19 different materials and finishes) were subjected to the aging method and then tested according to MIL-STD-202B, Method 208. Table I lists these and the results of the tests. Also part of this table is a comparison to the initial tests run on these samples[2]. Figs. 1 and 2 graphically show the evaluation results again, as compared to the initial results. (NC denotes no change).

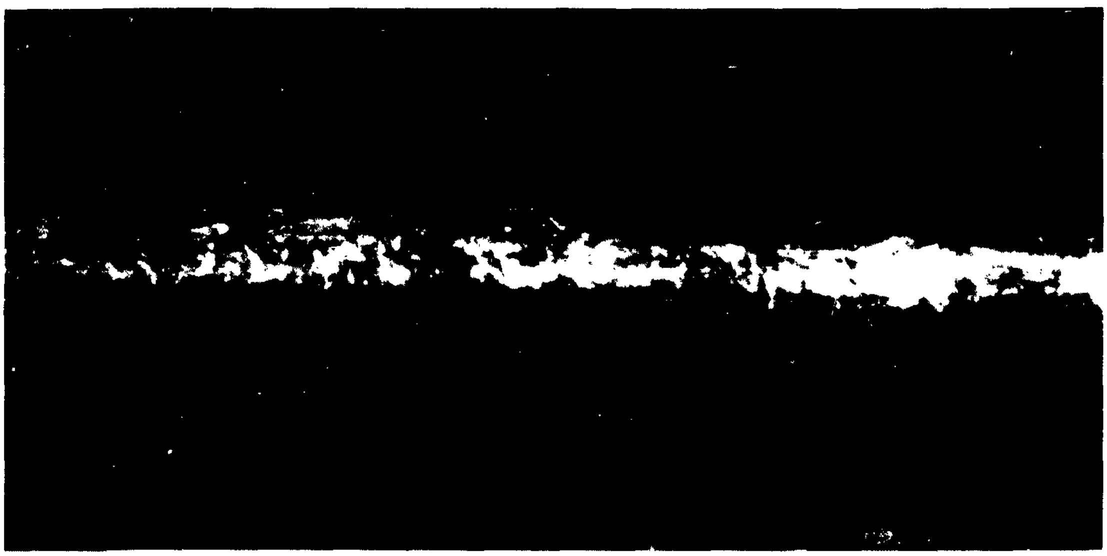
TABLE I

Boiling Water Aging

Sample #	Finish & Thickness (Microinch)	Base	Mil-Std-202, Method 208 Test Results		Comparison to Initial
1.	200 Sn	Copper	95-100%	Small on wet spots	down
2.	200 Sn flowed	Copper	98-100%	1 sample 80% unwet	down
5.	400 60/40	Copper	100%	Very tiny spots	N C
6.	95 Au	Copper	95-100%	Small spots of bare Au	up
7.	125 Au	Copper	down to 70%	Heavy dewet, some Au	
8.	70 Ni under 75 Au	Copper	$<50\%$	Heavy dewetting	N C
10.	280 Cu under 110 Sn	Brass	95-100%	Some unwet areas	down
11.	200 Cu under 400 60/40	Brass	100%	No defects	N C
12.	90 Au	Brass	95%	Some Dewet, some Au	up
14.	400 60/40	Brass	100%	No defects	N C
15.	320-400 Sn	Kovar	98-100%	Small unwet areas	N C
16.	400 60/40	Kovar	99-100%	Small unwet areas	down
18.	190 Au	Kovar	75%	Bare gold shows	up
19.	75 Ni under 0-60 Au	Kovar	85%	Bare gold, rough	up
21.	190 Au under 400 60/40	Kovar	75%	Dewetting	down
22.	40 Sn	Dumet	$<50\%$	Unwet areas	down
24.	75 Au	Dumet	98-100%	Some bare gold (2 tests)	up
29.	110 Au	Nickel	50%	Heavy dewetting 10-15% bare	up
30.	110 Au under 400 60/40	Nickel	99-100%	Slight dewetting	N C

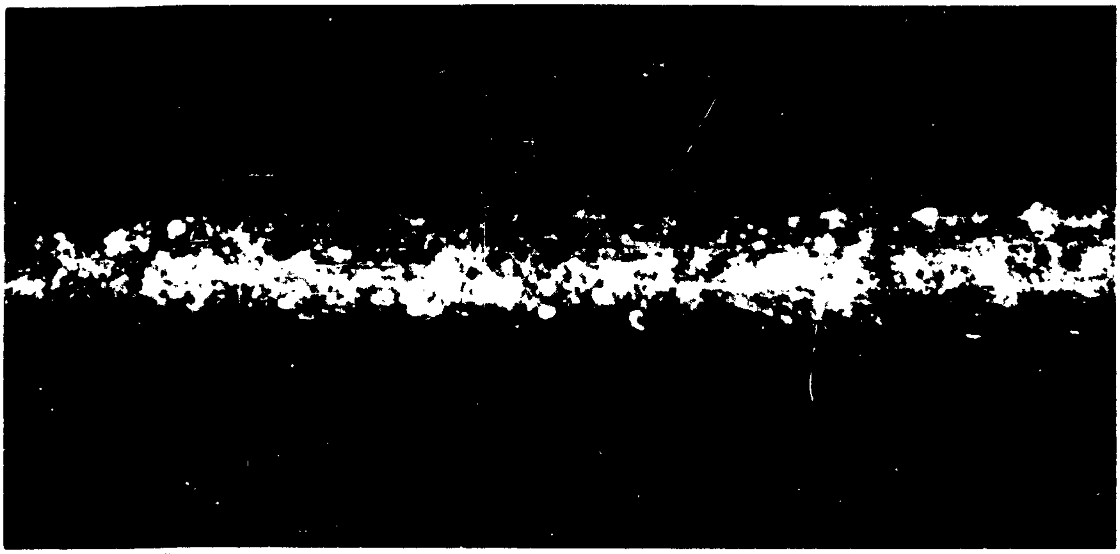


#24 - Initial

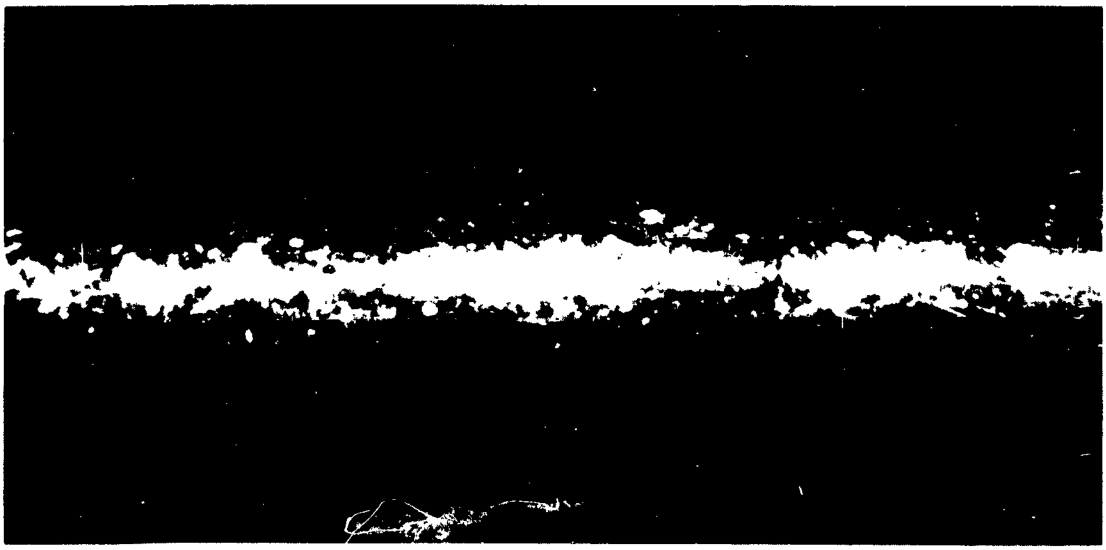


#24 - Boiling Water Aged

Fig. 1



#21 - Initial



#21 - Boiling Water Aged

Fig. 2

These results show that heavy tin or tin alloy finishes withstand the aging, thinner tin or tin alloy finishes are downgraded and gold plated finishes increase in solderability. The breakoff point has not yet been determined between heavy and thin tin finishes.

The latter is the reverse of what will happen during natural aging. It is assumed that the boiling water removes or changes the natural corrosion products or other surface contaminates which may have been present initially. This improvement happened in nearly every case of gold plated parts.

On this basis the method will probably be discounted as a possible aging method.

C. Heat Aging

This method of aging involves heating the parts to be tested in a hot air heated atmosphere. In this case temperatures of 200°, 250°, and 300°F were selected as a starting point. The means of heating was a forced circulating air furnace to insure even exposure. Length of exposure was one hour. Tables II, III, and IV list the samples and results. Figs. 3 and 4 graphically show the results.

Exposures to this test did not change the solderability of the samples. In a few isolated cases some changes were found but these could not be related to changes occuring during natural aging

At this point it is assumed that there will be some changes in nearly all of the natural aging samples so that again it appears that the heat aging method will be discounted.

From the results that were obtained at Battelle Memorial Institute[1] and others it is likely that much longer exposure times (24 hours or greater) are necessary to produce significant changes. When there is a drop it is also reported that the defects do not resemble natural defects.

TABLE II

Heat Aging at 200°F

Sample #	Base	Finish & Thickness (Microinch)	Mil-Std-202B, Method 208 Test Results		Comparison to Initial
1	Copper	200 Sn	99-100%	1 large unwet area	N C
2	Copper	200 Sn flowed	98-100%	Small spots	N C
6	Copper	95 Au	90-95%	Bare gold shows	N C
8	Copper	70 Ni under 75 Au	50%	Very heavy dewetting	N C
14	Brass	400 60/40	99-100%	Very small spots	N C
16	Kovar	400 60/40	99-100%	Small spots	N C
17	Kovar	110 Au	90-95%	Dewetting, bare gold	N C
29	Nickel	110 Au	$<$ 50%	Bare gold, heavy dewet	N C
30	Nickel	110 Au under 400 60/40	99-100%	Few unwet spots	N C

TABLE III

Heat Aging at 250°F

Sample #	Base	Finish & Thickness (Microinch)	Mil-Std-202B, Method 208 Test Results	Comparison to Initial
1	Copper	200 Sn	99-100% except 1 lead showing large unwet area	N C
2	Copper	200 Sn flowed	8 show 99-100% - 2 show 80-90% unwet	N C
6	Copper	95 Au	90-95% bare gold, pinholes	N C
8	Copper	70 Ni under 75 Au	50-75% fairly heavy dewetting & bare	N C
14	Brass	400 60/40	99-100%	N C
16	Kovar	400 60/40	99-100%	N C
17	Kovar	110 Au	down to 85% bare gold some dewet	N C
29	Nickel	110 Au	$<$50% bare gold, heavy dewet	N C
30	Nickel	110 Au under 400 60/40	99-100%	N C

TABLE IV

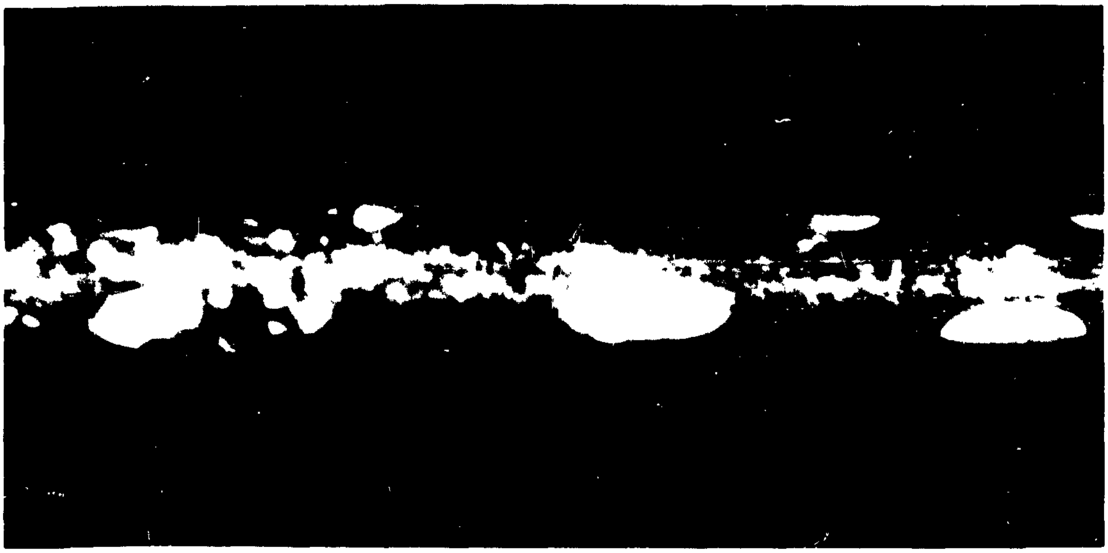
Heat Aging at 300°F

Sample #	Base	Finish & Thickness (Microinch)	Mil-Std-202B, Method 208 Test Results	Comparison to Initial
1	Copper	200 Sn	99-100%	N C
2	Copper	200 Sn flowed	99-100%	N C
6	Copper	95 Au	90-95% bare gold little dewetting	N C
8	Copper	70 Ni under 75 Au	down to 50% heavy dewet, some bare	N C
14	Brass	400 60/40	99-100%	N C
16	Kovar	400 60/40	99-100% some small pinholes	N C
17	Kovar	110 Au	95% bare gold, no dewetting	N C
29	Nickel	110 Au	down to 75% some bare Au, heavy dewetting	N C
30	Nickel	110 Au under 400 60/40	99-100%	N C

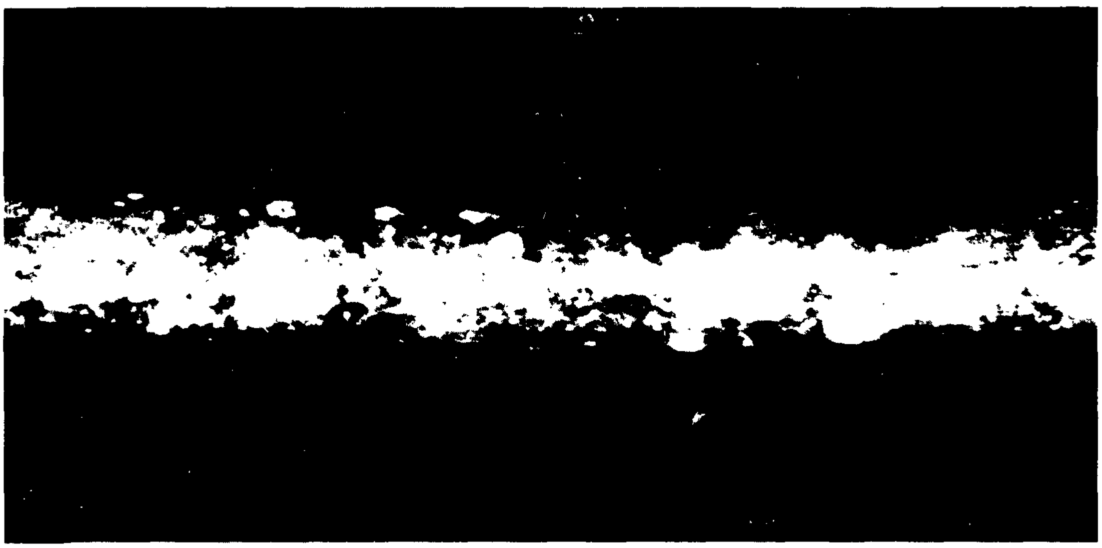
#17 - Initial

#17 - Heat Aged 200°

Fig. 3



#17 - Heat Aged 250°



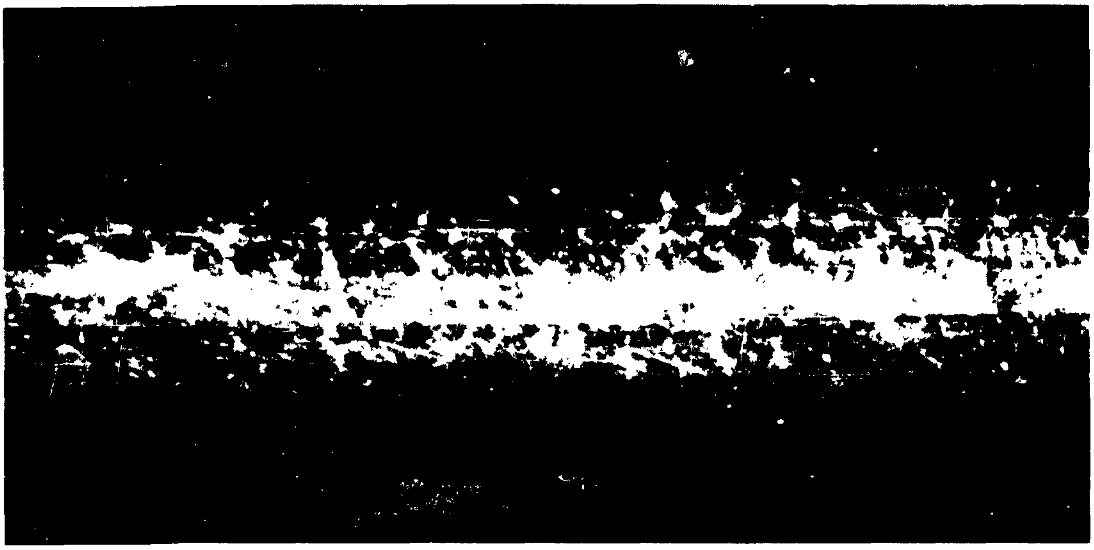
#17 - Heat Aged 300°

Fig. 3 (continued)

#1 - Initial

#1 - Heat Aged 200°

Fig. 4



#1 - Heat Aged 250°

#1 - Heat Aged 300°

Fig. 4 (continued)

D. Steam Aging

This method is a new approach to the problem. It is not believed that any prior experimentation has been done. The basis of the method is to subject the parts to an extreme of what is found in the atmosphere. The two major items which are significant are oxygen and water vapor (humidity). Using heat as a means to increase the reaction rates the samples are subjected for a short time to the extreme atmosphere.

The actual procedure used to create the above conditions was to suspend the samples over boiling distilled water. The container is a 2000 ml beaker covered with stainless steel plates such that the ambient temperature over the water is 200-210°F. Also the plates are arranged in such a way that air is free to enter the container. The time of exposure to this environment was two hours.

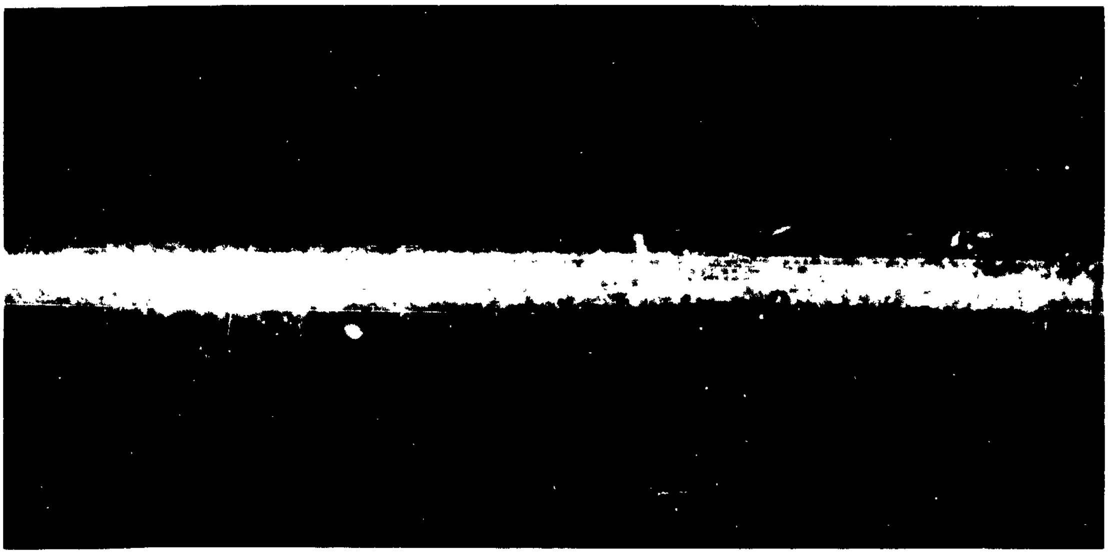
The results of this method are shown in Table V and Figs. 5 and 6. It was found that this method downgraded the majority of the samples to some extent. All those except one which showed no change were hot solder dipped finishes. The exception was a gold plated unit showing good initial solderability. In those cases where the samples were downgraded the defects appeared to be natural appearing. At this point in the testing it looks as though this method has much promise.

To extend the practical use of the steam aging method, further work is planned for an evaluation of solderability after exposure of test parts to standard test methods such as that found in Mil-Std-202 (Method 103A).

TABLE V

Steam Aging

Sample #	Base	Finish & Thickness (Microinch)	Mil-Std-202B, Method 208 Test Results	Comparison to Initial
1	Copper	200 Sn	99-100%	N C
2	Copper	200 Sn flowed	95-100% unwet area	down
5	Copper	400 60/40	99-100%	N C
7	Copper	125 Au	90% 1 almost total-unwet	down
9	Brass	400 Sn (discontinuous)	99-100%	N C
10	Brass	280 Cu under 110 Sn	85-90% large unwet spots	down
13	Brass	400 Sn flowed	$\sim$ 90% unwet areas	down
18	Kovar	190 Au	50% bare Au and dewet	down
21	Kovar	190 Au under 400 60/40	$\sim$ 75% heavily dewet	down
25	Dumet	Flash Ni under Au	90-95% bare gold, very little dewet	N C
29	Nickel	110 Au	$<$ 50% bare gold, rough, heavy dewet	down
30	Nickel	110 Au under 400 60/40	95-100% unwet areas - lumps	N C



#10 - Initial

#10 - Steam Aged

Fig. 5

#7 - Initial

#7 - Steam Aged

Fig. 6

Part II - Solderability of Lugs, Stranded Wire, etc.

A. General

The work during this quarter has been confined to investigation of test methods which involve the flowing of a preform on the part to be tested. There are a number of methods for doing this on flat samples[3,4] but only one has been attempted to date on small parts such as terminations. This test was described in last quarter's report[1]. In all methods attempted, the same preform was used but the methods of heating were different.

B. Flux Bath Method

This test consists of dipping the part to be tested which has been previously wrapped in a continuous spiral with a solder preform (10 mil 63/37 tin-lead wire), into a flux bath. The flux bath used was a solution of water white rosin and polyethylene glycol 400 heated to 450°F.

Preliminary tests using this method indicated that it might be a good one. Known unsolderable surfaces caused the preform to ball up or even fall off. Solderable surfaces allowed the preform to flow out showing good wetting. Figs. 7 and 8 show these conditions and the results of this test for terminals and stranded wire respectively.

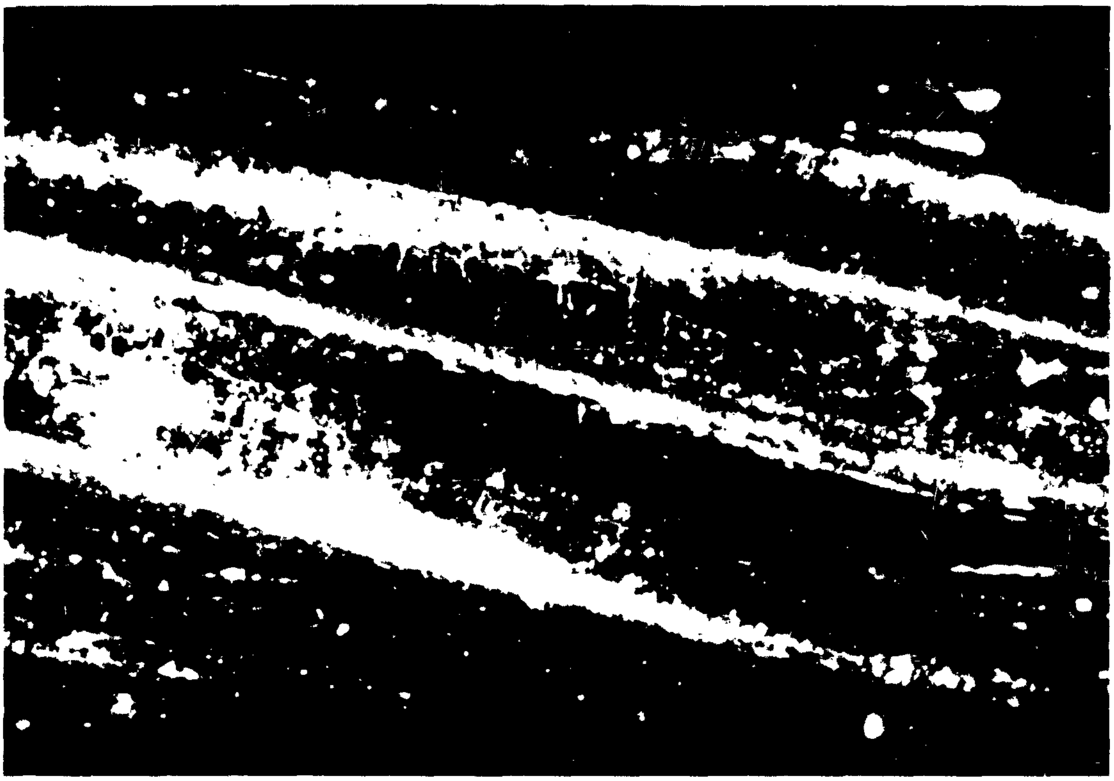
There are a number of advantages to a test of this type. In all cases the .010 inch diameter wire is adaptable to application to a sample. This includes everything from pigtail leads to complex shapes such as solder lugs. Also it is non-destructive with regards to the part or component.

Evaluation of the results of a test of this type could be made quantitative or at least semi-quantitative so that most of the human judgment is removed.

Solderability tested - flux bath method.

Stranded wire shows partial wetting.

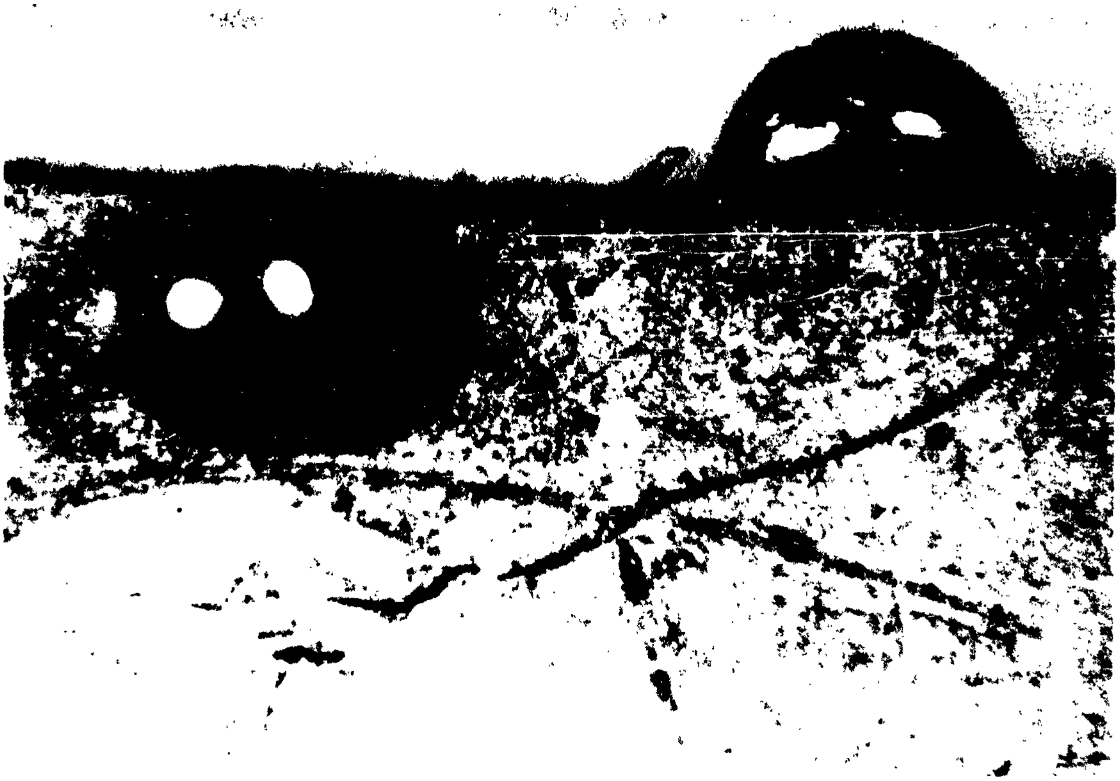
Fig. 7



Solderability tested - Mil-Std-202, Method 208

Stranded wire shows good wetting except on tops of strands.

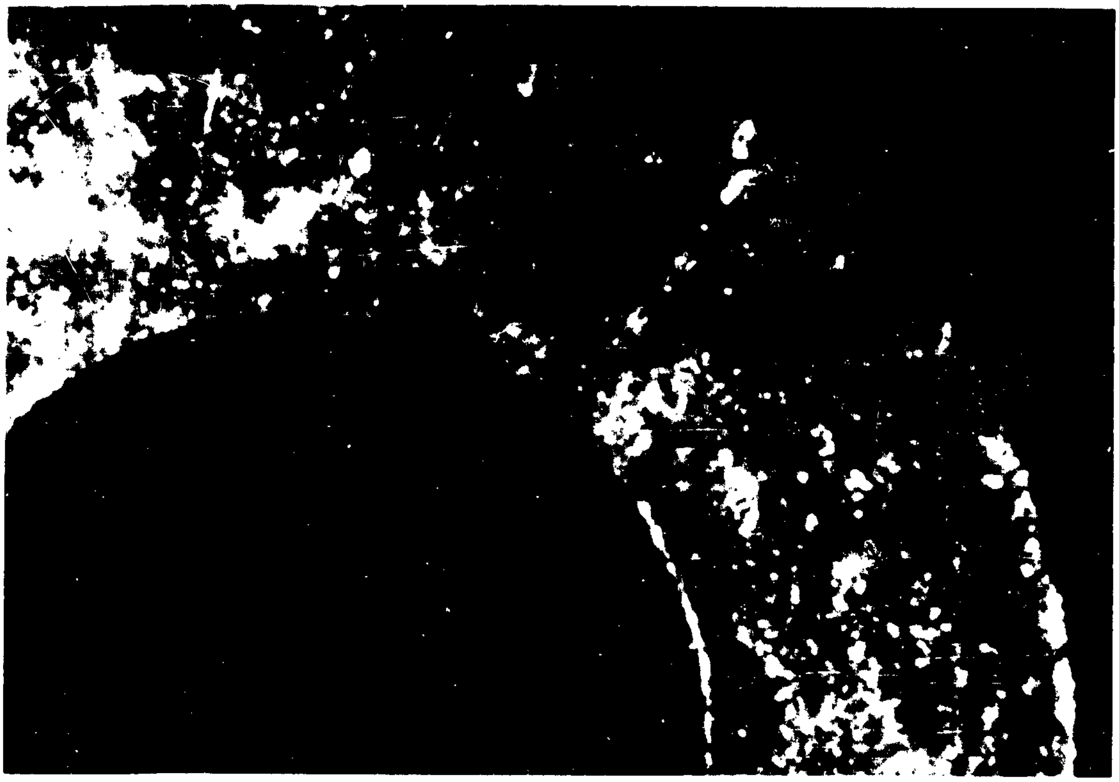
Fig. 7 (continued)



Solderability tested - flux bath method.

Terminal shows very poor wetting.

Fig. 8



Solderability tested - flux bath method.

Terminal shows good wetting.

Fig. 8 (continued)

One method of doing this would be to evaluate the shape of the flowed solder and compare it to standard drawings or photographs. RCA does this with the new test they have developed[2]. Another method which would work is to evaluate the angle of the flowed solder and surface of the test piece. When the solder preform balls up a negative angle would be produced. When the preform flows out a very low positive angle is produced. Two other methods of evaluation that would work with this type of test would be an actual measurement of the spread of the solder on the surface and a measure of the continuity of the solder after flowing - the more continuous the greater the solderability.

The real advantage of using a flux bath rather than an inert bath is that pre-cleaning of the solder wire and other such precautions need not be taken. Since the flux does the cleaning at the time of solder flowing these variables are eliminated. Also, a fluxing condition similar to what a part would receive in an actual soldering situation is built into the test.

However, during the course of investigation some difficulties were encountered, one of which appears to be unsurmountable. The first difficulty encountered was smoking of the heated flux bath. In general, the greater the percentage of rosin in the bath the greater the volume of smoke emitted. This is not a serious problem as any sort of simple hood will carry the vapors away as they are not what would be considered excessive.

The second and more serious problem showed up during a series of experiments to determine proper dwell time in the bath. The bath being used at the time was 50% rosin and 50% polyethylene glycol 400. First noted was the manner in which the preform melted on the part. Melting begins at the free end or smallest section of the sample (depending on sample

type and shape) and progresses towards the larger end or held end. If it is a component for example, melting will begin at end of the lead and progress towards the body. This progression takes place at a slow rate. It was also observed that the rate was slower on copper and faster on a material such as kovar. The second observation was the length of time from immersion into the bath to the start of preform flow. On a .032" dia. copper wire this was 8 - 9 seconds. Combining the initial heat up time and the flow time the situation is as follows. For complete flow of the preform on a .032" dia. copper wire the time required is 12 seconds. For a #14 stranded copper wire this time becomes a minute.

At this point it was decided to investigate the heat transfer properties of the system. Table VI gives the thermal properties for copper, kovar, rosin and polyethylene glycol 400 at 450°F. It can be seen that the bath has low thermal conductivity and high specific heat whereas the metallic materials are just the opposite. This indicates that very poor heat transfer ability exists in the direction needed. Investigation of other materials to improve the heat transfer has proved fruitless. It seems that no materials exist that will improve the situation and still passes the chemical properties required - inert or nearly so and miscible with rosin.

There are two possibilities of improving the heat transfer with the existing bath. One is to provide turbulent flow in the vicinity of the sample. However, turbulent flow has the problem of dislodging the solder on the surfaces of samples which have poor solderability. The second is to preheat the sample. Here again there is a problem of oxidation of the surfaces and necessarily elaborate equipment for its prevention.

With these facts it was decided to stop active work on this approach for the time being at least.

TABLE VI

Thermal Properties of Various Materials at 450°F

Material	Specific Heat g-cal/g	Thermal Conductivity g-cal/(sec.)(cm2)(°c/cm)
Copper	.093	.92
Kovar	.[illegible]30	.046
Polyethylene Glycol (400)	.60	.0006
* Rosin	> .60	< .0006

* Note: Data for rosin are unavailable in standard literature, however, experience shows these values go in the directions indicated with regards to Polyethylene Glycol 400.

C. Resistance Heating

This method was attempted as a means of supplying heat to the sample. The procedure involved is to wrap the part to be tested with the 10 mil solder wire, clamp it between two terminals and pass electrical current through it. Due to the resistance of the material heat is generated.

Immediately after working with a few samples it became evident that problems existed. First the electrical resistance of the common materials vary widely. Secondly, a part of non-uniform section size will heat quite unevenly. Therefore, the method of heating was discounted on this basis.

D. Induction Heating

This also was considered as a method of heating the sample in order to flow a wrapped preform as described under B and C. The equipment used was a five kilowatt high frequency heater by General Electric.

The procedure used was to wrap the preform around the sample, apply the flux, insert in the coil, and apply the power. Again, as in resistance heating, the controlling factors are part size (mass) and electrical resistivity.

From a few experiments attempted it was found that control of the heat and uniformity of the heat were good. However, power requirements are a problem. For small wires of kovar or similar materials the power levels and times were very reasonable. However, for a large copper lug or stranded wires, nearly the whole 5 kw were required to do the job in 10 seconds. Since the cost of equipment capable of these power levels would be prohibitive, it was decided to suspend experimentation in this direction.

CONCLUSIONS

Part I

It is concluded that of the three artificial aging methods investigated, the steam method appears to be the most promising. Theoretically, it combines both humidity and oxygen as aging agents with heat as the accelerating agent. This method is being held as a possible test until it can be evaluated against the natural aging samples.

It is also concluded that the boiling water or hot air do not appear to meet the requirements of an accelerated aging method. The boiling distilled water is discounted on the basis of the increase in solderability shown on gold plated parts. Hot air has been set aside for the present on the basis of no effect using short exposure times and appearance of unnatural defect types after exposure.

Part II

It is concluded that the flux bath method for checking solderability meets all the requirements of a good solderability test except one. This exception is the heat transfer properties of the bath which, in this case, are poor. Since no substitute materials are available to improve the situation, the investigation was postponed. A search for a substitute material is, however, being continued. If this difficulty could be surmounted, it is felt that a good test could be developed. The ease of evaluation and the possibility of a quantitative evaluation are the major advantages of this method.

Both resistance and induction heating methods have been discounted on the basis of the great variances in size, section and electrical resistivity. It is concluded that a means of external heating must be used to minimize the effects of the above listed properties.

PROGRAM FOR NEXT QUARTER

Part I

During the next quarter a set of natural aging samples will be evaluated. This will cover a period of three months aging. Also to be evaluated for their aging characteristics will be atmospheres containing sulphate and sulphide producing elements. This is on the basis that all natural atmospheres contain these elements.

Part II

During the next quarter we will continue evaluation of methods to flow preforms using external heating and fluxing to provide the proper flow conditions.

IDENTIFICATION OF KEY PERSONNEL

J. DeVore, Project Supervisor	279	man-hours.
L. Zakraysek, Engineer	79	man-hours.
D. Blackwood, Metallographer	60	man-hours.

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